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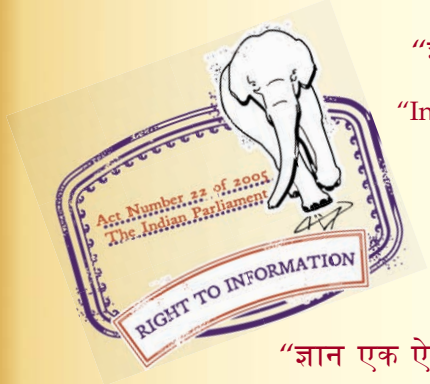

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IS : 8812 (Part I) - 1978
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METHODS OF CHEMICAL ANALYSIS OF HARD SOLDERS FOR JOINTING ALUMINIUM AND ALUMINIUM ALLOYS

PART I DETERMINATION OF SILVER, COPPER, ZINC, ANTIMONY, ARSENIC, IRON AND BISMUTH

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Indian Standard

METHODS OF CHEMICAL ANALYSIS OF HARD SOLDERS FOR JOINTING ALUMINIUM AND ALUMINIUM ALLOYS

PART I DETERMINATION OF SILVER, COPPER, ZINC, ANTIMONY, ARSENIC, IRON AND BISMUTH

0. FOREWORD

0.1 This Indian Standard (Part I) was adopted by the Indian Standards Institution on 26 June 1978, after the draft finalized by the Methods of Chemical Analysis Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 Chemical analysis of hard solders for jointing aluminium and aluminium alloys, the chemical composition of which is specified in IS : 5479-1969*, is covered in two parts of this standard. In this part, methods for determination of all constituents except aluminium in hard solders are covered. The method for determination of aluminium will be covered in Part II of this standard.

0.3 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960†.

1. SCOPE

1.1 This standard (Part I) covers the methods of determination of silver, copper, zinc, antimony, arsenic, iron and bismuth in hard solders, the chemical composition of which is given in IS : 5479-1969*.

2. SAMPLING

2.1 Laboratory Sample — It shall be drawn and prepared in accordance with IS : 1817-1961‡.

*Specification for solders for jointing aluminium and aluminium alloys.
†Rules for rounding off numerical values (*revised*).
‡Methods of sampling non-ferrous metals for chemical analysis.

3. QUALITY OF REAGENTS

3.1 Unless otherwise specified, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in the tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

4. DETERMINATION OF SILVER BY GRAVIMETRIC METHOD

4.1 Outline of the Method — The sample is dissolved in nitric acid and silver is precipitated as silver chloride by means of hydrochloric acid. The precipitate is filtered, washed, dried and weighed.

4.2 Reagents

4.2.1 *Tartaric Acid* — solid.

4.2.2 *Dilute Nitric Acid* — 1 : 1 and 1 : 99 (*v/v*).

4.2.3 *Dilute Hydrochloric Acid* — 1 : 9 (*v/v*).

4.3 Procedure

4.3.1 Transfer 1 g of an accurately weighed sample to a 250-ml beaker. Add 1 g of tartaric acid and 15 ml of dilute nitric acid (1 : 1). Heat gently to dissolve and boil to remove the brown fumes present. Dilute to about 100 ml.

4.3.2 Add sufficient amount of dilute hydrochloric acid slowly with constant stirring till the precipitation is complete and a few millilitre in excess. Let it stand for about 2 hours on a hot-plate to keep it warm.

4.3.3 Filter through a weighed Gooch crucible or sintered glass crucible No. 3 and wash several times with dilute nitric acid (1 : 99) till the washings are free from chloride and finally give two washings with warm water. Transfer the filtrate and washings to a 400-ml beaker and preserve for the determination of zinc.

4.3.4 Dry the precipitate at 120 to 130°C for about an hour. Cool in a desiccator to room temperature and weigh.

4.4 Calculation

$$\text{Silver, percent} = \frac{A}{B} \times 75{\cdot}26$$

where

A = mass in g of silver chloride, and

B = mass in g of sample taken.

*Specification for water for general laboratory use (*second revision*).

5. DETERMINATION OF COPPER BY ELECTROLYTIC METHOD (WHEN COPPER IS AN ALLOYING ELEMENT AND SILVER IS ABSENT)

5.1 Outline of the Method — After dissolution of the sample in dilute nitric acid followed by fuming with concentrated sulphuric acid, copper is precipitated alongwith arsenic, antimony and bismuth by passing hydrogen sulphide gas. The precipitate is dissolved in dilute nitric acid. Arsenic, antimony and bismuth are removed by ammonium hydroxide precipitation and copper in the filtrate is estimated electrolytically.

5.2 Apparatus — The platinum electrodes conforming to IS : 6882-1973* shall be used.

5.3 Reagents

5.3.1 *Dilute Nitric Acid* — 1 : 1 (*v/v*).

5.3.2 *Concentrated Sulphuric Acid* — rd = 1·84 (conforming to IS : 266-1961†).

5.3.3 *Hydrogen Sulphide* — gas.

5.3.4 *Hydrogen Sulphide Wash Solution* — Saturate dilute sulphuric acid (2 : 98) with hydrogen sulphide.

5.3.5 *Ferric Nitrate Solution* — 3 percent.

5.3.6 *Dilute Ammonium Hydroxide* — 1 : 1.

5.3.7 *Dilute Sulphuric Acid* — 1 : 5 (*v/v*).

5.3.8 *Urea* — solid.

5.3.9 *Ethanol or Methanol* — 95 percent (*v/v*).

5.4 Procedure

5.4.1 Dissolve 1 g of an accurately weighed sample in 15 ml of dilute nitric acid, add 5 ml of concentrated sulphuric acid and evaporate to fumes.

5.4.2 Cool, dilute to 150 ml, warm and pass a rapid steam of hydrogen sulphide gas for 15 minutes until all copper is precipitated.

5.4.3 Filter the solution through a No. 41 Whatman filter paper with paper pulp and wash the precipitate with hydrogen sulphide wash solution 6 to 7 times. Preserve the filtrate and washings for the estimation of zinc.

*Specification for platinum electrodes.
†Specification for sulphuric acid (*revised*).

5.4.4 Dissolve the precipitate in the original beaker with dilute nitric acid and wash the filter paper thoroughly with hot water. Add 5 ml of concentrated sulphuric acid and evaporate to fumes. Precipitate and separate copper according to **5.4.2** and **5.4.3**. Combine filtrate and washing with those preserved in **5.4.3**, boil off hydrogen sulphide and preserve the solution for the estimation of zinc.

5.4.5 Ignite the paper and precipitate in a porcelain crucible at 500°C. Transfer the ignited residue to the original beaker and rinse the crucible with a small amount of dilute nitric acid. Add 10 ml of dilute nitric acid and boil for 5 minutes. Add 50 ml of water, 1 ml of ferric nitrate solution and make ammoniacal with dilute ammonium hydroxide. Add 10 ml in excess. Boil for 2 to 3 minutes and allow to settle for half an hour. Filter and wash with hot water. Preserve the filtrate and washings.

5.4.6 Dissolve the precipitated ferric hydroxide in dilute nitric acid and precipitate iron with ammonium hydroxide and filter following the procedure described in **5.4.5**. Reject the precipitate.

5.4.7 Combine the filtrates and washings with the solution preserved in **5.4.5**, acidify with dilute nitric acid and then add 10 ml of dilute nitric acid and 10 ml of dilute sulphuric acid.

5.4.8 Weigh the cathode, adjust the electrodes in the solution and cover with a pair of split watch-glass. Add 2 g of urea. Electrolyze at a current density of 3 to 4 A/dm^2 for 45 minutes. When the solution becomes colourless reduce the current density to 0·3 A/dm^2 and continue electrolysis until the deposition of copper is complete as indicated by failure of copper to plate on the newly exposed cathode surface when the solution level is raised.

5.4.9 Without interrupting the current, lower the beaker slowly while rinsing the electrodes with water and collecting the washings in the electrolyte. Remove the cathode quickly, rinse it with water and then dip it in two successive baths of ethanol or methanol. Dry the cathode in an air oven at 110°C for 3 to 5 minutes, cool and reweigh the cathode. The difference in mass gives the mass of copper deposited.

5.5 Calculation

$$\text{Copper, percent} = \frac{A}{B} \times 100$$

where

A = mass in g of copper deposited, and

B = mass in g of sample taken.

6. DETERMINATION OF ZINC BY EDTA METHOD

6.1 Outline of the Method — Zinc is estimated by titration with EDTA with eriochrome black-T as indicator after separating all the other metals present.

6.2 Reagents

6.2.1 *Ammonium Chloride* — solid.

6.2.2 *Methyl Red Indicator Solution*

6.2.3 *Ammonium Hydroxide Solution* — 1 : 1 (*v/v*).

6.2.4 *Ammonium Chloride Wash Solution* — 2 percent.

6.2.5 *Ferrichloride Solution* — 5 percent.

6.2.6 *Dilute Hydrochloric Acid* — 1 : 1 (*v/v*).

6.2.7 *Concentrated Nitric Acid* — rd = 1·42 (conforming to IS : 264-1968*).

6.2.8 *Potassium Cyanide Solution* — 10 percent.

6.2.9 *Buffer Solution* — Dissolve 54 g of ammonium chloride in 300 ml of water, add 350 ml of ammonium hydroxide, and dilute to 1 litre. This solution has a *p*H of 10.

6.2.10 *Eriochrome Black-T Indicator Solution* — Dissolve 0·4 g of eriochrome black-T in 20 ml of ethanol, add 30 ml of triethanolamine and store in a polyethylene dropping bottle.

6.2.11 *Sodium Salt of EDTA Standard Solution (0·05 M)* — Dissolve 18·6 g of the salt in 600 ml of water and dilute to 1 litre with water. Standardize this solution against standard zinc solution (*see* **6.2.12**) and find out the equivalent of the EDTA solution in terms of g of zinc per millilitre of solution.

6.2.12 *Standard Zinc Solution* — Dissolve 1·000 g of pure zinc in 50 ml of sulphuric acid (1 : 4). Cool to room temperature and dilute to 1 litre. One millilitre of this solution is equivalent to 1 mg of zinc (Zn).

6.2.13 *Formaldehyde* — 1 : 9 (*v/v*).

6.3 Procedure

6.3.1 Reduce the volume of the solution preserved either in **4.3.3** or in **5.4.5** to about 100 ml.

*Specification for nitric acid (*first revision*).

6.3.2 Add 5 to 7 g of ammonium chloride and 1 to 2 drops of methyl red indicator solution neutralize with dropwise addition of ammonium hydroxide solution till the indicator colour changes (red to yellow), add 2 to 3 drops more and boil for 5 minutes. Allow the precipitate to settle for half an hour and then filter and wash with ammonium chloride wash solution (6 to 7 washings) and preserve the filtrate.

NOTE—For solution in **4.3.3**, add 1 ml of ferric chloride solution before the addition of ammonium chloride and ammonium hydroxide solution.

6.3.3 For zinc-aluminium alloys, dissolve 0·5 g sample in 15 ml of dilute hydrochloric acid, adding a few drops of concentrated nitric acid towards the end. Boil off nitrous fumes, cool to room temperature, and make up to 250 ml in a volumetric flask. Mix thoroughly and pipette out 25 ml in a 250-ml beaker, and proceed as in **6.3.2**.

6.3.4 Dissolve the precipitate in hot dilute hydrochloric acid and reprecipitate aluminium again, filter and wash as described in **6.3.2**. Reject the precipitate. Combine filtrate and washings with filtrate and washings as preserved in **6.3.2**.

6.3.5 Dilute the solution to about 500 ml in a 1-litre beaker, add 10 ml of potassium cyanide solution and 125 ml of buffer solution. Add 5 to 6 drops of eriochrome black-T indicator and sufficient formaldehyde solution, just give a red colour (30 to 40 ml solution is sufficient).

6.3.6 Titrate slowly with EDTA solution to a blue-green end point. Add 5 ml of formaldehyde solution, and if the blue-green colour changes to red, titrate again with EDTA solution to the blue-green end point. Continue the formaldehyde addition and EDTA titrations until the addition of formaldehyde has no effect within 2 minutes on the blue-green end point.

6.4 Calculation

$$\text{Zinc, percent} = \frac{A \times B}{C} \times 100$$

where

A = volume in ml of EDTA solution required for titration of the solution,

B = EDTA equivalent in terms of g of zinc per millilitre, and

C = mass in g of sample represented in the aliquot used.

7. DETERMINATION OF ANTIMONY BY POTASSIUM BROMATE METHOD

7.1 Procedure is same as described in IS : 1940-1969*, only dissolution of the sample is to be done in dilute nitric acid and tartaric acid (1 : 1) for

*Methods of chemical analysis of tin ingot (*first revision*).

silver containing alloys, silver separated as silver chloride and solution to be fumed with sulphuric acid. For other alloys dissolution is to be done in dilute hydrochloric acid (1 : 1) adding 1 to 2 ml concentrated nitric acid in the end and then fuming with sulphuric acid.

8. DETERMINATION OF ARSENIC BY MOLYBDENUM BLUE (PHOTOMETRIC) METHOD

8.1 Procedure is same as described in IS : 1940-1969*.

9. DETERMINATION OF COPPER BY CUPRIC BROMIDE (PHOTOMETRIC) METHOD (WHEN COPPER IS AN IMPURITY)

9.1 Procedure is same as described in IS : 1940-1969*, only dissolution is to be done in dilute nitric acid (1 : 1). For silver-containing alloy, silver should be removed as silver chloride and solution evaporated to syrupy consistency, for other alloys dissolution is to be done in dilute hydrochloric acid (1 : 1), adding 1-2 ml of concentrated nitric acid towards the end and evaporated to syrupy consistency.

10. DETERMINATION OF IRON BY ORTHO-PHENANTHROLINE (PHOTOMETRIC) METHOD

10.1 Procedure is same as described in IS : 1940-1969*.

11. DETERMINATION OF BISMUTH BY THIOUREA (PHOTOMETRIC) METHOD

11.1 Procedure is same as described in IS : 1940-1969*, only dissolution is to be done as in **9.1**.

*Methods of chemical analysis of tin ingot (*first revision*).

(*Continued from page* 2)

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